Analytical Laboratory

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Order Summary Report

Order Number: **J13110386**

Project Name: Frontier Sampling - Wed

Customer Name(s): Bill Kennedy, Wayne Chapman

Customer Address: 3195 Pine Hall Rd
Mailcode: Belews Steam Station
Belews Creek, NC 28012

Lab Contact: Jason C Perkins Phone: 980-875-5348

Report Authorized By: (Signature) _____ **Date:** 12/13/2013
Jason C Perkins

Program Comments:

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

Data Flags & Calculations:

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

Certification:

The Analytical Laboratory holds the following State Certifications : North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID's & Descriptions:

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2013028500	BELEWS	20-Nov-13 7:30 AM	P. GASSETT	FGD Purge Eff
2013028501	BELEWS	20-Nov-13 7:35 AM	P. GASSETT	EQ Tank Eff
2013028502	BELEWS	20-Nov-13 7:40 AM	P. GASSETT	BIOREACTOR 1 INF
2013028503	BELEWS	20-Nov-13 7:45 AM	P. GASSETT	BioReactor 2 Inf
2013028504	BELEWS	20-Nov-13 7:50 AM	P. GASSETT	BioReactor 2 Eff
2013028506	BELEWS	20-Nov-13	P. GASSETT	FILTER BLANK
2013028507	BELEWS	11-Nov-13 1:45 PM	D. Baker	METALS TRIP BLANK

7 Total Samples

Technical Validation Review

Checklist:

COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures).	☑ Yes	☐ No
All Results are less than the laboratory reporting limits.	☐ Yes	☑ No
All laboratory QA/QC requirements are acceptable.	☑ Yes	☐ No

Report Sections Included:

- ☑ Job Summary Report
- ☑ Sample Identification
- ☑ Technical Validation of Data Package
- ☑ Analytical Laboratory Certificate of Analysis
- ☐ Analytical Laboratory QC Report
- ☑ Sub-contracted Laboratory Results
- ☐ Customer Specific Data Sheets, Reports, & Documentation
- ☐ Customer Database Entries
- ☑ Chain of Custody
- ☑ Electronic Data Deliverable (EDD) Sent Separatel

Reviewed By: DBA Account Date: 12/13/2013

Certificate of Laboratory Analysis

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Order # J13110386

Site: FGD Purge Eff — **Sample #: 2013028500**

Collection Date: 20-Nov-13 7:30 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC								
Bromide	**71**	mg/L		5	50	EPA 300.0	11/22/2013 14:31	JAHERMA
Chloride	**6500**	mg/L		100	1000	EPA 300.0	11/22/2013 14:31	JAHERMA
MERCURY (COLD VAPOR) IN WATER								
Mercury (Hg)	**129**	ug/L		5	100	EPA 245.1	11/26/2013 13:18	DKJOHN2
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	**203**	mg/L		0.5	10	EPA 200.7	12/02/2013 13:04	MHH7131
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	**347**	ug/L		10	10	EPA 200.8	12/09/2013 12:46	DJSULL1
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	**211**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Cadmium (Cd)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Chromium (Cr)	**273**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Copper (Cu)	**123**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Nickel (Ni)	**230**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Selenium (Se)	**2540**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Silver (Ag)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Thallium (Tl)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Vanadium (V)	**212**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
Zinc (Zn)	**259**	ug/L		10	10	EPA 200.8	12/06/2013 13:13	DJSULL1
SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)								
Vendor Parameter	**Complete**					Vendor Method		V_AS&C
TOTAL DISSOLVED SOLIDS								
TDS	**19000**	mg/L		250	1	SM2540C	11/25/2013 10:28	DSBAKE1
TOTAL SUSPENDED SOLIDS								
TSS	**2500**	mg/L		250	1	SM2540D		TJA7067

Site: EQ Tank Eff — **Sample #: 2013028501**

Collection Date: 20-Nov-13 7:35 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY (COLD VAPOR) IN WATER								
Mercury (Hg)	**91.0**	ug/L		2.5	50	EPA 245.1	11/26/2013 13:20	DKJOHN2
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	**203**	mg/L		0.5	10	EPA 200.7	12/02/2013 13:08	MHH7131

Certificate of Laboratory Analysis

Order # J13110386

Site: EQ Tank Eff **Sample #: 2013028501**

Collection Date: 20-Nov-13 7:35 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	**159**	ug/L		10	10	EPA 200.8	12/09/2013 12:50	DJSULL1
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	**179**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Cadmium (Cd)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Chromium (Cr)	**230**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Copper (Cu)	**104**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Nickel (Ni)	**196**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Selenium (Se)	**2140**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Silver (Ag)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Thallium (Tl)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Vanadium (V)	**178**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1
Zinc (Zn)	**219**	ug/L		10	10	EPA 200.8	12/06/2013 13:17	DJSULL1

Site: BIOREACTOR 1 INF **Sample #: 2013028502**

Collection Date: 20-Nov-13 7:40 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
NITRITE + NITRATE (COLORIMETRIC)								
Nitrite + Nitrate (Colorimetric)	**9.5**	mg-N/L		0.25	25	EPA 353.2	11/22/2013 12:02	BGN9034
INORGANIC IONS BY IC								
Bromide	**69**	mg/L		5	50	EPA 300.0	11/22/2013 14:50	JAHERMA
Chloride	**6800**	mg/L		100	1000	EPA 300.0	11/22/2013 14:50	JAHERMA
Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)								
Vendor Parameter	**Complete**	ug/l				Vendor Method		V_AS&C
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	**185**	mg/L		0.5	10	EPA 200.7	12/02/2013 13:12	MHH7131
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	**105**	ug/L		10	10	EPA 200.8	12/09/2013 13:02	DJSULL1

Certificate of Laboratory Analysis

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Order # J13110386

Site: BIOREACTOR 1 INF **Sample #: 2013028502**

Collection Date: 20-Nov-13 7:40 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Cadmium (Cd)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Chromium (Cr)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Copper (Cu)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Nickel (Ni)	**10.0**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Selenium (Se)	**131**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Silver (Ag)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Thallium (Tl)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Vanadium (V)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
Zinc (Zn)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:20	DJSULL1
SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)								
Vendor Parameter	**Complete**					Vendor Method		V_AS&C
TOTAL DISSOLVED SOLIDS								
TDS	**15000**	mg/L		25	1	SM2540C	11/25/2013 10:28	DSBAKE1
TOTAL SUSPENDED SOLIDS								
TSS	**< 5**	mg/L		5	1	SM2540D		TJA7067

Site: BioReactor 2 Inf **Sample #: 2013028503**

Collection Date: 20-Nov-13 7:45 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)								
Vendor Parameter	**Complete**	ug/l				Vendor Method		V_AS&C
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	**187**	mg/L		0.5	10	EPA 200.7	12/02/2013 13:16	MHH7131
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Cadmium (Cd)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Chromium (Cr)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Copper (Cu)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Nickel (Ni)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Selenium (Se)	**27.2**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Silver (Ag)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Thallium (Tl)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Vanadium (V)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1
Zinc (Zn)	**< 10**	ug/L		10	10	EPA 200.8	12/06/2013 13:24	DJSULL1

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Order # J13110386

Site: BioReactor 2 Inf — **Sample #: 2013028503**
Collection Date: 20-Nov-13 7:45 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst

Site: BioReactor 2 Eff — **Sample #: 2013028504**
Collection Date: 20-Nov-13 7:50 AM — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
NITRITE + NITRATE (COLORIMETRIC)								
Nitrite + Nitrate (Colorimetric)	**< 0.01**	mg-N/L		0.01	1	EPA 353.2	11/22/2013 12:03	BGN9034
INORGANIC IONS BY IC								
Bromide	**71**	mg/L		5	50	EPA 300.0	11/22/2013 15:09	JAHERMA
Chloride	**6800**	mg/L		100	1000	EPA 300.0	11/22/2013 15:09	JAHERMA
Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)								
Vendor Parameter	**Complete**	ug/l				Vendor Method		V_AS&C
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	**197**	mg/L		0.5	10	EPA 200.7	12/02/2013 13:20	MHH7131
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Cadmium (Cd)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Chromium (Cr)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Copper (Cu)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Nickel (Ni)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Selenium (Se)	**7.24**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Silver (Ag)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Thallium (Tl)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Vanadium (V)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
Zinc (Zn)	**< 5**	ug/L		5	5	EPA 200.8	12/06/2013 13:27	DJSULL1
SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)								
Vendor Parameter	**Complete**					Vendor Method		V_AS&C

Site: FILTER BLANK — **Sample #: 2013028506**
Collection Date: 20-Nov-13 — Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	**< 1**	ug/L		1	1	EPA 200.8	12/09/2013 12:55	DJSULL1

Certificate of Laboratory Analysis

Order # J13110386

Site: METALS TRIP BLANK **Sample #: 2013028507**

Collection Date: 11-Nov-13 1:45 PM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS BY ICP								
Boron (B)	**< 0.05**	mg/L		0.05	1	EPA 200.7	12/02/2013 12:56	MHH7131
TOTAL RECOVERABLE METALS BY ICP-MS								
Arsenic (As)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Cadmium (Cd)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Chromium (Cr)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Copper (Cu)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Nickel (Ni)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Selenium (Se)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Silver (Ag)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Thallium (Tl)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Vanadium (V)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1
Zinc (Zn)	**< 1**	ug/L		1	1	EPA 200.8	12/06/2013 13:06	DJSULL1

APPLIED SPECIATION AND CONSULTING, LLC

18804 Northcreek Parkway Bothell, WA, 98011
Tel: (425) 483-3300 Fax: (425) 483-9818
www.appliedspeciation.com

December 6, 2013

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078
(704) 875-5245

Project: Frontier Pilot (Belews Creek) WWTS (Wed Sampling) (LIMS# J13110386)

Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for total mercury, hexavalent chromium, and selenium speciation analyses on November 21, 2013. The samples were received in a sealed cooler at 0.1°C on November 22, 2013. Mercury quantitation was performed via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS). Hexavalent chromium analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Any issues associated with the analyses are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Jeremy Maute
Project Coordinator
Applied Speciation and Consulting, LLC

Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Frontier Pilot (Belews Creek) WWTS (Wed Sampling) (LIMS# J13110386)

December 6, 2013

1. Sample Reception

Three (3) aqueous samples were submitted for hexavalent chromium and selenium speciation analyses on November 21, 2013. Three (3) additional samples were submitted for total mercury quantitation. All samples were received in acceptable condition on November 22, 2013 in a sealed container at 0.1°C.

All samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. The 40mL borosilicate glass vials submitted for total mercury were preserved with bromine monochloride (BrCl) solution. The resulting samples were stored in a secure polyethylene container, known to be free from trace metals contamination, until the analyses could be performed.

An aliquot of each sample submitted for hexavalent chromium analysis was filtered (0.45um) and stored in a secure refrigerator maintained at a temperature of 4^{o}C, until the analyses could be performed.

An aliquot of each sample requiring selenium speciation evaluation was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

Total Mercury Quantitation by CV-ICP-MS All samples and preparation blanks for total mercury quantitation were preserved with 2% (v/v) BrCl. The resulting samples were analyzed for mercury via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS).

Hexavalent Chromium Analysis by IC-ICP-DRC-MS Prior to analysis, all samples were injected directly into autosampler vials. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

Selenium Speciation Analysis by IC-ICP-CRC-MS Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

Total Mercury Quantitation by CV-ICP-MS The sample fractions for total mercury quantitation were analyzed by cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS) on November 25, 2013. Aliquots of each sample are reacted with a reductant in-line and transported to a gas-liquid separator. The volatile elemental mercury that is formed is then swept by a stream of argon gas into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and separated on the basis of their mass-to-charge ratio (m/z) by a mass spectrometer. A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Hexavalent Chromium Analysis by IC-ICP-DRC-MS Each sample for hexavalent chromium analysis was analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on December 5, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The

eluting chromium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

Selenium Speciation Analysis by IC-ICP-CRC-MS Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on December 4, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL values for methylseleninic acid and selenomethionine are calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

The eMDL values for hexavalent chromium and mercury are calculated using the standard deviation of the preparation blanks preserved and analyzed concurrently with the submitted samples.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Jeremy Maute
Project Coordinator
Applied Speciation and Consulting, LLC

Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy
Project Name: Frontier Pilot (Belews Creek) WWTS (Wed Sampling)
Contact: Jay Perkins
LIMS #J13110386

Date: December 6, 2013
Report Generated by: Jeremy Maute
Applied Speciation and Consulting, LLC

Sample Results

Sample ID	Total Hg	Cr(VI)	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Unknown Se Species (n)
FGD Purge Eff	NR	ND (< 1.1)	327	48.1	12.6	ND (< 4.5)	ND (< 4.5)	0 (0)
BioReactor 1 Inf	0.0887	ND (< 1.1)	70.8	30.0	ND (< 0.73)	1.9	ND (< 1.1)	0 (0)
BioReactor 2 Inf	0.0245	NR	NR	NR	NR	NR	NR	NR
BioReactor 2 Eff	0.0060	ND (< 1.1)	ND (< 1.8)	ND (< 0.86)	ND (< 0.73)	ND (< 1.1)	ND (< 1.1)	0 (0)

All results reflect the applied dilution and are reported in µg/L
NR = Analysis not requested
ND = Not detected at the applied dilution
SeCN = Selenocyanate
MeSe(IV) = Methylseleninic acid
SeMe = Selenomethionine
Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS
n = number of unknown Se species observed

Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy
Project Name: Frontier Pilot (Belews Creek) WWTS (Wed Sampling)
Contact: Jay Perkins
LIMS #J13110386

Date: December 6, 2013
Report Generated by: Jeremy Maute
Applied Speciation and Consulting, LLC

Quality Control Summary - Preparation Blank Summary

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 5x	eMDL 250x	eMDL 1000x
Hg	-0.0003	-0.0005	-0.0001	0.0002	-0.0002	0.0003	0.0002	0.0009	-	-
Cr(VI)	-0.4	-1.0	-0.2	-0.2	-0.5	0.4	0.001	-	-	1.1
Se(IV)	0.00	0.00	0.00	0.00	0.00	0.00	0.007	-	1.8	7.2
Se(VI)	0.00	0.00	0.00	0.00	0.00	0.00	0.003	-	0.86	3.5
SeCN	0.00	0.00	0.00	0.00	0.00	0.00	0.003	-	0.73	2.9
MeSe(IV)	0.00	0.00	0.00	0.00	0.00	0.00	0.005	-	1.1	4.5
SeMe	0.00	0.00	0.00	0.00	0.00	0.00	0.005	-	1.1	4.5

eMDL = Estimated Method Detection Limit

*Please see narrative regarding eMDL calculations

Quality Control Summary - Certified Reference Materials

Analyte (µg/L)	CRM	True Value	Result	Recovery
Hg	NIST 1641d	1568	1635	104.3
Cr(VI)	LCS	1.000	1.161	116.1
Se(IV)	LCS	4.79	4.71	98.5
Se(VI)	LCS	4.74	4.59	96.8
SeCN	LCS	4.46	4.35	97.6
MeSe(IV)	LCS	3.24	3.05	94.2
SeMe	LCS	4.66	4.48	96.1

Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy
Project Name: Frontier Pilot (Belews Creek) WWTS (Wed Sampling)
Contact: Jay Perkins
LIMS #J13110386

Date: December 6, 2013
Report Generated by: Jeremy Maute
Applied Speciation and Consulting, LLC

Quality Control Summary - Matrix Duplicates

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Hg	Batch QC	0.0151	0.0151	0.0151	0.4
Cr(VI)	Batch QC	ND (< 1.1)	ND (< 1.1)	NC	NC
Se(IV)	BioReactor 2 Eff	ND (< 1.8)	ND (< 1.8)	NC	NC
Se(VI)	BioReactor 2 Eff	ND (< 0.86)	ND (< 0.86)	NC	NC
SeCN	BioReactor 2 Eff	ND (< 0.73)	ND (< 0.73)	NC	NC
MeSe(IV)	BioReactor 2 Eff	ND (< 1.1)	ND (< 1.1)	NC	NC
SeMe	BioReactor 2 Eff	ND (< 1.1)	ND (< 1.1)	NC	NC

ND = Not detected at the applied dilution
NC = Value was not calculated due to one or more concentrations below the eMDL

Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Hg	Batch QC	2.000	2.273	112.9	2.000	2.210	109.8	2.8
Cr(VI)	Batch QC	1000	860.1	86.0	1000	845.7	84.6	1.7
Se(IV)	BioReactor 2 Eff	1390	1297	93.3	1390	1290	92.8	0.6
Se(VI)	BioReactor 2 Eff	1261	1221	96.8	1261	1201	95.3	1.6
SeCN	BioReactor 2 Eff	1144	1074	93.9	1144	1054	92.2	1.9

CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd
Huntersville, N. C. 28078
(704) 875-5245
Fax: (704) 875-4349

Analytical Laboratory Use Only

ORDER# 3131O386 | MATRIX: OTHER | Samples Originating From: NC ✓ SC ___
Logged By: D.B. | Date & Time: 11/21/13 1030 | SAMPLE PROGRAM: Ground Water ___ NPDES ___ Drinking Water ___ UST ___ RCRA Waste ___
Vendor: AS&C | PO#ISW01.1894 | Cooler Temp (C): 0.5
Vendor: | MR #:

[19]Page 1 of 2
DISTRIBUTION
ORIGINAL to LAB,
COPY to CLIENT

Customer must Complete

1)Project Name: Frontier Pilot (Belews Creek) WWTS (Wed Sampling)		2)Phone No:	
2) Client: Bill Kennedy, Wayne Chapman		4)Fax No:	
5)Business Unit: 20006	6)Process: SFHDW1205	Mail Code:	
8)Oper. Unit: FHGO	9)Res. Type: 3500	10)Reso. Center: 1830000	

Customer to complete all appropriate non-shaded areas.

[15]Preserv.: 1=HCl, 2=H_2SO_4, 3=HNO_3, 4=Ice, 5=None

[16]Analyses Required

Sampling conducted: 2nd and 4th Wednesday

LAB USE ONLY [11]Lab ID	Se Speciation Bottle ID	[13]Sample Description or ID	Date	Time	Signature	[17]Comp.	[18]Grab	TDS, TSS	Br, Cl (Dionex)	Metals* + Hg**	Se (IMS), filtered	Hg 200.8 (V_AS&C)	C-NO2-NO3	Cr, Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies)
20130208500		FGD Purge Eff	11-20-13	7:30	R Gossett			1	1	1	1			1
8504		EQ Tank Eff.		7:35						1	1			
8502		BioReactor 1 Inf		7:40				1	1	1**	1	1	1	1
8503		BioReactor 2 Inf		7:45						1**		1		
8504		BioReactor 2 Eff		7:50					1	1**		1	1	1
DS 8505		~~Bio Pilot Eff~~			Not Running									
		~~FE Pilot Eff~~			Not Running									
8506		Filter Blk									1			
8507		Metals Trip Blk	11/21/13	1345	D Baker					1**				

AS&C charge the Cr and Se speciation charges to PO Frontier Water. **Duke is not paying for these costs**

Filtering of the Se is performed in the field please provide a filter blank too.

Return kit to Travis Thornton

Customer to complete appropriate columns to right

Customer to sign & date below - fill out from left to right.

1) Relinquished By: R Gossett	Date/Time: 11-20-2013 14:38	2) Accepted By:	Date/Time: 11/21/13 10:29
3) Relinquished By:	Date/Time: 11/21/13 1200	4) Accepted By: FEDEX	Date/Time: 11/21/13
5)Relinquished By:	Date/Time:	6)Accepted By:	Date/Time:
7)Relinquished By:	Date/Time:	8)Accepted By:	Date/Time:
9)Seal/Locked By:	Date/Time:	10) Seal/Lock Opened By:	Date/Time:
11)Seal/Locked By:	Date/Time:	12)Seal/Lock Opened By:	Date/Time:

Comments:

Customer, IMPORTANT! Please indicate desired turnaround.

[22]Requested Turnaround
14 Days 12/4/13
*7 Days ___
- 48 Hr ___
*Other ___
* Add. Cost Will Apply

* B by TRM/ICP As, Cd, Cr, Cu, Ni, Se, Ag, Zn, V, Tl by TRM/IMS 1**=No Hg

Rec'd by Aubrey Ernst 11/22/13 15:50 @ 0.1°C

CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

[19]Page 1 of 2

DISTRIBUTION
ORIGINAL to LAB,
COPY to CLIENT

Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd
Huntersville, N. C. 28078
(704) 875-5245
Fax: (704) 875-4349

Analytical Laboratory Use Only

ORDER# 3131O386 | MATRIX: OTHER | Samples Originating From NC ✓ SC ___
Logged By D.B. | Date & Time 11/21/13 1030
SAMPLE PROGRAM: Water ___ Ground ___ NPDES ___ Drinking Water ___ UST ___ RCRA Waste ___
Vendor AS&C
PO#ISW01.1894
Cooler Temp (C) 0.5
Vendor:

[15]Preserv.: 1=HCL 2=H_2SO_4 3=HNO_3 4=Ice 5=None

Customer must Complete

1)Project Name	Frontier Pilot (Belews Creek) WWTS (Wed Sampling)	2)Phone No:	
2) Client:	Bill Kennedy, Wayne Chapman	4)Fax No:	
5)Business Unit:	20006	6)Process: SFHDW1205	Mail Code:
8)Oper. Unit:	FHGO	9)Res. Type: 3500	10)Reso. Center: 1830000

MR #

Customer to complete all appropriate non-shaded areas.

Sampling conducted: 2nd and 4th Wednesday

Customer to complete appropriate columns to right

LAB USE ONLY [11]Lab ID	Se Speciation Bottle ID	[13]Sample Description or ID	Date	Time	Signature	[17]Comp.	[18]Grab	TDS, TSS	Br, Cl (Dionex)	Metals* + Hg**	Se (IMS), filtered	Hg 200.8 (V_AS&C)	C-NO2-NO3	Cr, Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies)
						[16]Analyses Required								4
20130285 00		FGD Purge Eff	11-20-13	7:30	Phil Cossett			1	1	1	1			1
8504		EQ Tank Eff.		7:35						1	1			
8502		BioReactor 1 Inf		7:40				1	1	1**	1	1	1	1
8503		BioReactor 2 Inf		7:45						1**		1		
8504		BioReactor 2 Eff		7:50					1	1**		1	1	1
~~DB 8505~~		~~Bio Pilot Eff~~			Not Running				1	1**	1	1	1	1
0		~~mF Pilot Eff~~			Not Running			1	1	1**	1	1	1	1
8506		Filter Blk									1			
8507		Metals Trip Blk	11/1/13	1345	D. Baker					1**				

AS&C charge the Cr and Se speciation charges to PO Frontier Water Duke is not paying for these costs

Filtering of the Se is performed in the field please provide a filter blank too.

Return kit to Travis Thornton____

Customer to sign & date below - fill out from left to right.

1) Relinquished By: Phil Cossett	Date/Time: 11-20-2013 14:38	2) Accepted By:	Date/Time: 11/21/13 10:29
3) Relinquished By:	Date/Time: 11/21/13 1200	4) Accepted By: FEDEX	Date/Time: 11/21/13
5)Relinquished By	Date/Time	6)Accepted By:	Date/Time
7)Relinquished By	Date/Time	8)Accepted By:	Date/Time
9)Seal/Locked By	Date/Time	10) Seal/Lock Opened By	Date/Time
11)Seal/Locked By	Date/Time	12)Seal/Lock Opened By	Date/Time

Comments:

* B by TRM/ICP As, Cd, Cr, Cu, Ni, Se, Ag, Zn, V, Tl by TRM/IMS 1**=No Hg

Customer, IMPORTANT! Please indicate desired turnaround.

[22]Requested Turnaround

14 Days _____
*7 Days _____
*48 Hr _____
*Other _____
* Add. Cost Will Apply